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=> d que 110  
L4 3493 SEA OXIDAT? AND HYDROPEROX? AND ((ALKYL OR METHYL OR ETHYL OR  
PROPYL OR BUTYL OR ISOPROPYL OR ISOBUTYL) (W) BENZENE OR CUMENE

OR METHYLBENZENE OR TOLUENE OR ETHYLBENZENE OR ALKYL BENZEN? OR  
PROPYLBENZEN? OR PROPYLBENZEN? OR ISOPROPYLBENZEN? OR ISOBUTYLB  
ENZEN?)

L5 159 SEA L4 AND (AMMONIA OR BASE)  
L6 20 SEA L5 AND NEUTRAL?  
L10 16 DUP REM L6 (4 DUPLICATES REMOVED)

=> d l10 ibib abs hitind 1-16

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L10 ANSWER 1 OF 16 HCAPLUS COPYRIGHT 2005 ACS on STN DUPLICATE 1

ACCESSION NUMBER: 2004:681442 HCAPLUS

DOCUMENT NUMBER: 141:192260

TITLE: Oxidation process for producing  
hydroperoxides using neutralizing  
base

INVENTOR(S): Yang, Jiemin; Black, Jesse Raymond

PATENT ASSIGNEE(S): USA

SOURCE: U.S. Pat. Appl. Publ., 8 pp.

CODEN: USXXCO

DOCUMENT TYPE: Patent

LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 2004162448	A1	20040819	US 2004-761641	20040121
US 2004236152	A1	20041125	US 2004-761676	20040121
WO 2004074230	A1	20040902	WO 2004-US4009	20040211
W:	AE, AE, AG, AL, AL, AM, AM, AM, AT, AT, AU, AZ, AZ, BA, BB, BG, BG, BR, BR, BW, BY, BY, BZ, BZ, CA, CH, CN, CN, CO, CO, CR, CR, CU, CU, CZ, CZ, DE, DE, DK, DK, DM, DZ, EC, EC, EE, EE, EG, ES, ES, FI, FI, GB, GD, GE, GE, GH, GM, HR, HR, HU, HU, ID, IL, IN, IS, JP, JP, KE, KE, KG, KG, KP, KP, KR, KR, KZ, KZ, KZ, LC, LK, LR, LS, LS, LT, LU, LV, MA, MD, MD, MG, MK, MN, MW, MX, MX, MZ, MZ, NA, NI			
RW:	BW, GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG			
WO 2004074241	A1	20040902	WO 2004-US4010	20040211
W:	AE, AE, AG, AL, AL, AM, AM, AM, AT, AT, AU, AZ, AZ, BA, BB, BG, BG, BR, BR, BW, BY, BY, BZ, BZ, CA, CH, CN, CN, CO, CO, CR, CR, CU, CU, CZ, CZ, DE, DE, DK, DK, DM, DZ, EC, EC, EE, EE, EG, ES, ES, FI, FI, GB, GD, GE, GE, GH, GM, HR, HR, HU, HU, ID, IL, IN, IS, JP, JP, KE, KE, KG, KG, KP, KP, KR, KR, KZ, KZ, KZ, LC, LK, LR, LS, LS, LT, LU, LV, MA, MD, MD, MG, MK, MN, MW, MX, MX, MZ, MZ, NA, NI			
RW:	BW, GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, HU, IE, IT, LU, MC, NL, PT, RO, SE, SI, SK, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG			

## PRIORITY APPLN. INFO.:

US 2003-447526P

P 20030214

US 2004-761641

A 20040121

## OTHER SOURCE(S):

CASREACT 141:192260

AB A process for **oxidn.** of **alkylbenzenes** to produce **hydroperoxides** comprises: providing an **oxidn.** feed consisting essentially of an organic phase, the **oxidn.** feed comprising one or more **alkylbenzenes** and a quantity of **neutralizing base** having a pH of from about 8 to about 12.5 in 1 to 10% aqueous solution, the quantity of **neutralizing base** being effective to **neutralize** at least a portion of acids formed during the **oxidn.**, the **oxidn.** feed comprising up to an amount of water effective to increase **neutralization** of acids formed during the **oxidn.** without forming a sep. aqueous phase; exposing the **oxidn.** feed to **oxidn.** conditions effective to produce an **oxidn.** product stream comprising one or more product **hydroperoxides**.

IC ICM C07C409-00

INCL 568577000

CC 45-4 (Industrial Organic Chemicals, Leather, Fats, and Waxes)

ST **alkylbenzene oxidn hydroperoxide**  
**neutralizing base**IT **Oxidation**

(**oxidn.** process for producing **hydroperoxides** using **neutralizing base**)

IT **Hydroperoxides**

RL: IMF (Industrial manufacture); PREP (Preparation)

(**oxidn.** process for producing **hydroperoxides** using **neutralizing base**)

IT **Bases, reactions**

RL: RCT (Reactant); RACT (Reactant or reagent)

(**oxidn.** process for producing **hydroperoxides** using **neutralizing base**)

IT 80-15-9P, **Cumene hydroperoxide** 52208-72-7P,  
**sec-Butylbenzene hydroperoxide**

RL: IMF (Industrial manufacture); PREP (Preparation)

(**oxidn.** process for producing **hydroperoxides** using **neutralizing base**)

IT 98-82-8, **Cumene** 135-98-8 7664-41-7, **Ammonia**,  
reactions

RL: RCT (Reactant); RACT (Reactant or reagent)

(**oxidn.** process for producing **hydroperoxides** using **neutralizing base**)

L10 ANSWER 2 OF 16 HCAPLUS COPYRIGHT 2005 ACS on STN DUPLICATE 2

ACCESSION NUMBER: 2004:264594 HCAPLUS

DOCUMENT NUMBER: 140:287254

TITLE: Process for preparing enantiomerically pure  
(S)-3-hydroxy- $\gamma$ -butyrolactone from a D-hexose  
source.INVENTOR(S): Gurjar, Mukund Keshao; Kumar, Pradeep; Deshmukh, Anis  
Naim; Upadhyay, Rajesh Kumar; Upadhyay, Puspesh Kumar

PATENT ASSIGNEE(S): Council of Scientific and Industrial Research, India

SOURCE: U.S., 7 pp.

CODEN: USXXAM

DOCUMENT TYPE: Patent

LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 6713639	B1	20040330	US 2002-281615	20021028
PRIORITY APPLN. INFO.:			US 2002-281615	20021028

OTHER SOURCE(S): CASREACT 140:287254

AB A process for the preparation of enantiomerically pure (S)-3-hydroxy- $\gamma$ -butyrolactone (I) comprises dissolving a D-hexose source in an aqueous alkali solution, heating the solution to 40-50° for 1-4 h to obtain a dark yellow to dark red solution, adding a peroxide to the solution, raising the temperature of the solution to about 70° for 8-24 h to obtain a reaction mixture of 3,4-dihydroxybutyric acid and glycolic acid, cooling the reaction mixture to about 25°, adding an acid to the reaction mixture to about pH 1.0, evaporating the reaction mixture to dryness to remove H<sub>2</sub>O and glycolic acid to obtain a yellow syrup, **neutralizing** this with a solid **base**, extracting with an organic solvent, drying the organic layer over anhydrous Na<sub>2</sub>SO<sub>4</sub> to obtain a residue, and purifying the residue over a silica gel column using a mixture of organic solvents as an eluant. Thus, maltose monohydrate in 0.16 M NaOH was heated at 40° for 2h.; 80% **cumene hydroperoxide** was added slowly. The temperature was increased slowly to 70° and kept at this temperature for another 8 h. The reaction mixture was cooled to 25° and then to 0° and acidified with concentrate H<sub>2</sub>SO<sub>4</sub> to pH 1 followed by concentration to dryness

at

50° to remove glycolic acid and water. To the yellow colored syrup formed, ice was added followed by **neutralization** with solid NaHCO<sub>3</sub>, extraction with Et acetate, and drying over Na<sub>2</sub>SO<sub>4</sub>. The solvent was removed under reduced pressure and the residue was chromatographed on a silica gel column using EtOAc:Pet ether (4:6), to give pure I having an optical purity of 94% in 54% yield.

IC ICM C07D307-20

INCL 549313000

CC 27-5 (Heterocyclic Compounds (One Hetero Atom))

Section cross-reference(s): 33

ST hydroxybutyrolactone chiral prepn; maltose **oxidn** cyclization

IT Cyclization

**oxidation**

(preparation of enantiomerically pure (S)-3-hydroxy- $\gamma$ -butyrolactone from a D-hexose source)

IT 75-75-2, Methanesulfonic acid 75-91-2 80-15-9, **Cumene hydroperoxide** 144-55-8, Sodium bicarbonate, reactions 497-19-8, Sodium carbonate, reactions 1310-73-2, Sodium hydroxide, reactions 1493-13-6, Trifluoromethanesulfonic acid 7647-01-0, Hydrochloric acid, reactions 7664-93-9, Sulfuric acid, reactions  
 RL: RGT (Reagent); RACT (Reactant or reagent)  
 (preparation of enantiomerically pure (S)-3-hydroxy- $\gamma$ -butyrolactone from a D-hexose source)

REFERENCE COUNT: 27 THERE ARE 27 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L10 ANSWER 3 OF 16 HCAPLUS COPYRIGHT 2005 ACS on STN DUPLICATE 3

ACCESSION NUMBER: 2002:790254 HCAPLUS

DOCUMENT NUMBER: 137:296556

TITLE: Method and system for manufacturing **cumene hydroperoxide** by the peroxidation of **cumene**

INVENTOR(S): Fulmer, John William; Scott, Eugene Edward; Kight, William Dale

PATENT ASSIGNEE(S): General Electric Company, USA  
 SOURCE: U.S., 8 pp.  
 CODEN: USXXAM  
 DOCUMENT TYPE: Patent  
 LANGUAGE: English  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
US 6465695	B1	20021015	US 2001-916775	20010727
WO 2003011820	A1	20030213	WO 2002-US22083	20020609
WO 2003011820	C1	20031211		
W:	AE, AG, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ, DE, DK, DM, DZ, EC, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, OM, PH, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, UZ, VN, YU, ZA, ZM, ZW			
RW:	GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW, AM, AZ, BY, KG, KZ, MD, RU, TJ, TM, AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, TR, BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG			
EP 1414793	A1	20040506	EP 2002-752279	20020709
R:	AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, SI, LT, LV, FI, RO, MK, CY, AL, TR, BG, CZ, EE, SK			
US 2003092943	A1	20030515	US 2002-225095	20020821
US 6620974	B2	20030916		

PRIORITY APPLN. INFO.:  
 US 2001-916775 A 20010727  
 WO 2002-US22083 W 20020609

AB **Cumene hydroperoxide** is manufactured in high yield and selectivity by reacting **cumene** and oxygen in the presence of a water phase containing aqueous **ammonia**, and in the absence of an additive containing an alkali or alkaline earth metal, to form **cumene hydroperoxide**. A system for producing **cumene hydroperoxide** is described which comprises a **cumene feed** in fluid communication with a reactor having a **cumene hydroperoxide oxidate** outlet, an oxygen feed in fluid communication with the reactor, and an **ammonia** feed in fluid communication with the **cumene** feed and/or the reactor, where the **cumene** feed, the oxygen feed, the **ammonia** feed, and the reactor are free of an additive comprising an alkali or alkaline earth metal. Process flow diagrams are presented.

IC ICM C07C409-02

INCL 568571000

CC 45-4 (Industrial Organic Chemicals, Leather, Fats, and Waxes)  
 Section cross-reference(s): 25, 47, 48

ST **cumene hydroperoxide** manuf; peroxidn manuf  
**cumene hydroperoxide**

IT Peroxidation  
 (method and system for manufacturing **cumene hydroperoxide** by the peroxidn. of **cumene**)

IT Alkali metal hydroxides  
 Alkali metal salts  
 Alkaline earth hydroxides  
 Alkaline earth salts  
 RL: EPR (Engineering process); PEP (Physical, engineering or chemical process); RGT (Reagent); PROC (Process); RACT (Reactant or reagent)

(neutralizing agents; in manufacturing **cumene hydroperoxide** by the peroxidn. of **cumene**)

IT 80-15-9P, **Cumene hydroperoxide**  
RL: EPR (Engineering process); IMF (Industrial manufacture); PEP (Physical, engineering or chemical process); PREP (Preparation); PROC (Process)  
(method and system for manufacturing **cumene hydroperoxide** by the peroxidn. of **cumene**)

IT 98-82-8, **Cumene**  
RL: EPR (Engineering process); PEP (Physical, engineering or chemical process); RCT (Reactant); PROC (Process); RACT (Reactant or reagent)  
(method and system for manufacturing **cumene hydroperoxide** by the peroxidn. of **cumene**)

IT 7782-44-7, Oxygen, reactions  
RL: EPR (Engineering process); PEP (Physical, engineering or chemical process); RCT (Reactant); RGT (Reagent); PROC (Process); RACT (Reactant or reagent)  
(method and system for manufacturing **cumene hydroperoxide** by the peroxidn. of **cumene**)

IT 463-79-6D, Carbonic acid, Group IA or IIA carbonates, reactions  
497-19-8, Sodium carbonate, reactions 1336-21-6, Ammonium hydroxide  
7664-38-2D, Phosphoric acid, Group IA or IIA phosphates, reactions  
7664-41-7, **Ammonia**, reactions  
RL: EPR (Engineering process); PEP (Physical, engineering or chemical process); RGT (Reagent); PROC (Process); RACT (Reactant or reagent)  
(neutralizing agents; in manufacturing **cumene hydroperoxide** by the peroxidn. of **cumene**)

IT 7732-18-5; Water, processes  
RL: EPR (Engineering process); NUU (Other use, unclassified); PEP (Physical, engineering or chemical process); PROC (Process); USES (Uses)  
(solvent; method and system for manufacturing **cumene hydroperoxide** by the peroxidn. of **cumene**)

REFERENCE COUNT: 14 THERE ARE 14 CITED REFERENCES AVAILABLE FOR THIS RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L10 ANSWER 4 OF 16 HCAPLUS COPYRIGHT 2005 ACS on STN DUPLICATE 4

ACCESSION NUMBER: 1998:116085 HCAPLUS

DOCUMENT NUMBER: 128:129491

TITLE: Water-alkaline emulsion **cumene** oxidation process

INVENTOR(S): Zakoshansky, Vladimir Michailo; Griaznov, Andrei K.; Vasilieva, Irina Ivanovna; Fulmer, John William; Kight, William Dale

PATENT ASSIGNEE(S): General Electric Co., USA

SOURCE: Eur. Pat. Appl., 13 pp.

CODEN: EPXXDW

DOCUMENT TYPE: Patent

LANGUAGE: English

FAMILY ACC. NUM. COUNT: 1

PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
EP 816335	A1	19980107	EP 1997-304341	19970620
EP 816335	B1	20011031		
R: AT, BE, CH, DE, DK, ES, FR, GB, GR, IT, LI, LU, NL, SE, MC, PT, IE, FI				
US 5767322	A	19980616	US 1996-670304	19960627

ES 2163102	T3	20020116	ES 1997-304341	19970620
CN 1173491	A	19980218	CN 1997-114047	19970624
CN 1088059	B	20020724		
JP 10087609	A2	19980407	JP 1997-167816	19970625
RU 2183623	C2	20020620	RU 1997-111312	19970626
US 5908962	A	19990601	US 1998-20395	19980209

## PRIORITY APPLN. INFO.:

US 1996-670304

A 19960627

AB Greater efficiency in the title process using a cascade of reactors is obtained by splitting the reactor cascade into 2 stages with the 1st stage utilizing  $\text{NH}_4\text{NaCO}_3$  as the active carbonate in the stage containing  $\leq 18\%$  **cumene hydroperoxide** (I) and using  $\text{Na}_2\text{CO}_3$  as the active carbonate in the stage containing  $\geq 18\%$  I. By directly injecting **ammonia** into a recycle stream organic acids are efficiently **neutralized**. A counter current water wash of the 2nd stage also increases process efficiency by scrubbing out unwanted impurities. Control of pH in the process improves efficiency and reduces impurity levels.

IC ICM C07C409-10

CC 45-4 (Industrial Organic Chemicals, Leather, Fats, and Waxes)

Section cross-reference(s): 25

ST **cumene hydroperoxide** com prodn; wet **oxidn**  
process **cumene**; alk emulsion **oxidn cumene**;  
pH control **oxidn cumene**

IT pH

(control; water-alkaline emulsion **cumene oxidn**. process  
with good efficiency)

IT **Oxidation**

(water-alkaline emulsion **cumene oxidn**. process with  
good efficiency)

IT 497-19-8, Sodium carbonate, processes 94485-78-6, Carbonic acid ammonium  
sodium salt

RL: PEP (Physical, engineering or chemical process); PROC (Process)  
(**neutralizing** agent; water-alkaline emulsion **cumene**  
**oxidn**. process with good efficiency)

IT 80-15-9P, **Cumene hydroperoxide**

RL: IMF (Industrial manufacture); PREP (Preparation)  
(water-alkaline emulsion **cumene oxidn**. process with  
good efficiency)

IT 7664-41-7, **Ammonia**, processes

RL: PEP (Physical, engineering or chemical process); PROC (Process)  
(water-alkaline emulsion **cumene oxidn**. process with  
good efficiency)

IT 98-82-8, **Cumene**

RL: RCT (Reactant); RACT (Reactant or reagent)  
(water-alkaline emulsion **cumene oxidn**. process with  
good efficiency)

REFERENCE COUNT:

2

THERE ARE 2 CITED REFERENCES AVAILABLE FOR THIS  
RECORD. ALL CITATIONS AVAILABLE IN THE RE FORMAT

L10 ANSWER 5 OF 16 HCAPLUS COPYRIGHT 2005 ACS on STN

ACCESSION NUMBER: 1998:430270 HCAPLUS

DOCUMENT NUMBER: 129:82066

TITLE: Procedure for the extraction of **hydroperoxides**  
in the manufacture of resorcinol using substantially  
phenolic-free MIBK as the solvent

INVENTOR(S): Ohmae, Toshikazu; Tokumasu, Shigefumi; Ohki, Hideo

PATENT ASSIGNEE(S): Sumitomo Chemical Co., Ltd., Japan

SOURCE: Ger. Offen., 4 pp.

DOCUMENT TYPE: Patent  
 LANGUAGE: German  
 FAMILY ACC. NUM. COUNT: 1  
 PATENT INFORMATION:

PATENT NO.	KIND	DATE	APPLICATION NO.	DATE
DE 19756878	A1	19980625	DE 1997-19756878	19971219
JP 10175949	A2	19980630	JP 1996-340188	19961219
JP 3391644	B2	20030331		
US 5959155	A	19990928	US 1997-988182	19971210
BE 1011622	A3	19991109	BE 1997-1031	19971217
CN 1185434	A	19980624	CN 1997-108789	19971219
CN 1085202	B	20020522		

PRIORITY APPLN. INFO.: JP 1996-340188 A 19961219

AB Aqueous sodium hydroxide-containing solns. of **hydroperoxides** [e.g., 1,3-bis(2-**hydroperoxy**-2-propyl)benzene and 3-(2-hydroxy-2-propyl)-1-(2-**hydroperoxy**-2-propyl)benzene], formed by the **oxidn.** of 1,3-diisopropylbenzene in the manufacture of resorcinol, are extracted in high yield with a high phase-transfer efficiency by using MIBK containing ≤10 ppm of phenol or phenols as the extraction solvent. The separated **hydroperoxides** are then cleaved with acid, the MIBK solution **neutralized**, and then distilled or distilled and washed with an aqueous **base** to obtain the resorcinol (no data).

IC ICM C07C409-02  
 ICS C07C407-00; C07B063-00; B01D011-00

CC 35-2 (Chemistry of Synthetic High Polymers)  
 Section cross-reference(s): 25, 48

ST resorcinol manuf **hydroperoxide** extn MIBK; phase sepn MIBK **hydroperoxide** extn

IT **Hydroperoxides**  
 RL: PEP (Physical, engineering or chemical process); PUR (Purification or recovery); RCT (Reactant); PREP (Preparation); PROC (Process); RACT (Reactant or reagent)  
 (1,3-bis(2-**hydroperoxy**-2-propyl)benzene and 3-(2-hydroxy-2-propyl)-1-(2-**hydroperoxy**-2-propyl)benzene; procedure for extraction of **hydroperoxides** in the manufacture of resorcinol using substantially phenolic-free MIBK as the solvent)

IT Phase separation  
 (of **hydroperoxides** from aqueous NaOH solns. in the manufacture of resorcinol using substantially phenolic-free MIBK as the solvent)

IT Extraction  
 (of **hydroperoxides** in the manufacture of resorcinol using substantially phenolic-free MIBK as the solvent)

IT Phenols, processes  
 RL: PEP (Physical, engineering or chemical process); REM (Removal or disposal); PROC (Process); RACT (Reactant or reagent)  
 (procedure for extraction of **hydroperoxides** in the manufacture of resorcinol using substantially phenolic-free MIBK as the solvent)

IT 51985-06-9P  
 RL: BYP (Byproduct); RCT (Reactant); REM (Removal or disposal); PREP (Preparation); PROC (Process); RACT (Reactant or reagent)  
 (procedure for the extraction of **hydroperoxides** in the manufacture of resorcinol using substantially phenolic-free MIBK as the solvent)

IT 108-46-3P, Resorcinol, preparation



- RL: IMF (Industrial manufacture); PREP (Preparation).  
(procedure for the extraction of **hydroperoxides** in the manufacture of resorcinol using substantially phenolic-free MIBK as the solvent)
- IT 7732-18-5, Water, uses  
RL: NUU (Other use, unclassified); USES (Uses)  
(procedure for the extraction of **hydroperoxides** in the manufacture of resorcinol using substantially phenolic-free MIBK as the solvent)
- IT 108-10-1, MIBK  
RL: NUU (Other use, unclassified); PEP (Physical, engineering or chemical process); PROC (Process); USES (Uses)  
(procedure for the extraction of **hydroperoxides** in the manufacture of resorcinol using substantially phenolic-free MIBK as the solvent)
- IT 721-26-6P 13387-60-5P  
RL: PEP (Physical, engineering or chemical process); PUR (Purification or recovery); RCT (Reactant); PREP (Preparation); PROC (Process); RACT (Reactant or reagent)  
(procedure for the extraction of **hydroperoxides** in the manufacture of resorcinol using substantially phenolic-free MIBK as the solvent)
- IT 99-62-7, 1,3-Diisopropylbenzene 1310-73-2, Sodium hydroxide, reactions  
RL: RCT (Reactant); RACT (Reactant or reagent)  
(procedure for the extraction of **hydroperoxides** in the manufacture of resorcinol using substantially phenolic-free MIBK as the solvent)

L10 ANSWER 6 OF 16 HCAPLUS COPYRIGHT 2005 ACS on STN

ACCESSION NUMBER: 1970:31364 HCAPLUS

DOCUMENT NUMBER: 72:31364

TITLE: Formation of by-products during the **oxidation** of 1,1-diphenylethane and their influence on the autoxidation process

AUTHOR(S): Yurzhenko, T. I.; Dikii, M. A.; Vaida, M. S.

CORPORATE SOURCE: USSR

SOURCE: Usp. Khim. Org. Perekisnykh Soedin. Autookisleniya, Dokl. Vses. Konf., 3rd (1969), Meeting Date 1965, 365-70. Editor(s): Emanuel, N. M. Izd. "Khimiya": Moscow, USSR.  
CODEN: 21RAAM

DOCUMENT TYPE: Conference

LANGUAGE: Russian

- AB **Oxidn.** products of Ph<sub>2</sub>CHMe (I) (except the **hydroperoxide**) contain PhOH, BzOH, HCO<sub>2</sub>H, Ph<sub>2</sub>CO, AcPh, Ph<sub>2</sub>C(OH)Me, H<sub>2</sub>O<sub>2</sub>, H<sub>2</sub>O, and CO<sub>2</sub>. PhOH inhibits I autoxidn. Also, the acids inhibit I autoxidn., owing to their catalytic action on the decomposition of the **hydroperoxide** to PhOH. Carbonyl compds. and CO<sub>2</sub> are formed by an intramol. transformation of I peroxide radical and the decomposition of an acyl radical, resp. Thus, the autoxidn. of I and the formation of by-products may be represented by the following: I -O<sub>2</sub>→ Ph<sub>2</sub>CMeOO→ Ph<sub>2</sub>C OOMe→ Ph<sub>2</sub>CO + MeO; Ph<sub>2</sub>CMeOO-I→ Ph<sub>2</sub>CMeOOH-(-HO)→ Ph<sub>2</sub>CMeO-I→ Ph<sub>2</sub>CMeOH; Ph<sub>2</sub>CMeOOH-(-PhOH)→ BzMe-O<sub>2</sub>→ PhCOCH<sub>2</sub>OOH→ CH<sub>2</sub>O + BzOH; CH<sub>2</sub>O-O<sub>2</sub>→ HCO<sub>2</sub>H; BzOH→ PhCO<sub>2</sub>→ Ph + CO<sub>2</sub>. I should be oxidized in the presence of **bases** which **neutralize** PhOH and acids inhibiting it. Thus, Na<sub>2</sub>CO<sub>3</sub> increases the **hydroperoxide** yield to 60-5%. The deceleration of the autoxidn. of I (compared with **cumene**) is attributed to steric hindrance during the addition of the 2nd Ph radical at the α-C. The low rate of I autoxidn. may be addnl. explained by the smaller thermal stability of I **hydroperoxide**, which decomp. into by-products inhibiting the autoxidn.
- CC 25 (Noncondensed Aromatic Compounds)

IT **Oxidation**

(aut-, of diphenylethane, mechanism of)

L10 ANSWER 7 OF 16 HCAPLUS COPYRIGHT 2005 ACS on STN

ACCESSION NUMBER: 1954:28644 HCAPLUS

DOCUMENT NUMBER: 48:28644

ORIGINAL REFERENCE NO.: 48:5133b-i,5134a-i,5135a

TITLE: The mechanism of **oxidation**. IX.**Oxidation** and autoxidation of hydrazones

AUTHOR(S): Witkop, Bernhard; Kissman, Henry M.

CORPORATE SOURCE: Natl. Inst. of Health, Bethesda, MD

SOURCE: Journal of the American Chemical Society (1953), 75, 1975-80

CODEN: JACSAT; ISSN: 0002-7863

DOCUMENT TYPE: Journal

LANGUAGE: Unavailable

AB cf. *ibid.* 500; C.A. 47, 12400i. A reinvestigation of the spectrophotometric and chemical properties of the products of the BzO<sub>2</sub>H **oxidation** of PhCH:NNHPh (I) and p-MeOC<sub>6</sub>H<sub>4</sub>CH:NNHPh (II) led to their formulation as mixed aliphatic-aromatic azoxy compds. represented by RCH<sub>2</sub>N(O):NR' to which zwitterions, such as RCHN(OH):NR', and [RCH:N:NR']OH presumably contribute; the saltlike tautomers help to explain the high m.p., the stability, and the low solubility in nonpolar and polar solvents. The transformation of PhCH<sub>2</sub>N(O):NPh (III) with EtMgI to PhC(:NPh)NH<sub>2</sub> (IIIA) is formulated as a Stevens rearrangement and taken as circumstantial evidence for the location of the O at the N in the center of the triad. The reduction and rearrangement of hydrazone **hydroperoxides** is described and discussed and forms the basis for the proposal of a hypothetical biogenetic scheme for the naturally occurring aliphatic azoxy compound macrozamin. III, m. 203-6° (decomposition) (all m.p. are corrected), obtained in 49% yield by the method

of Bergmann (C.A. 17, 2418),  $\lambda_{\text{maximum}}$  252 m $\mu$  (log  $\epsilon$  3420) in EtOH, infrared absorption bands at 6.76, 7.62, 7.68, and 7.85 in Nujol. III in CHCl<sub>3</sub> turned from violet to yellow on heating and back to violet on cooling. The addition of Et<sub>2</sub>O to a colored solution of III in CHCl<sub>3</sub> discharged the color and precipitated a white solid, m. 201-3°, which did not give a violet color in CHCl<sub>3</sub> after treatment with Et<sub>2</sub>O or with a drop of H<sub>2</sub>O; it did not depress the decomposition point. of III. Treatment of CHCl<sub>3</sub> with a trace of MeONa prior to the addition of III prevented the color formation. III gave also highly colored solns. in glacial AcOH which turned brown on heating, but did not change back to the original red-violet on cooling. III treated with wet Et<sub>2</sub>O did not give colored solns. in glacial AcOH. II (4.52 g.) and 3.30 g. BzO<sub>2</sub>H in 60 cc. Et<sub>2</sub>O refrigerated 3 days, and the yellow crystalline deposit washed with Et<sub>2</sub>O and recrystd. from EtOAc gave 2.6 g. (54%) p-MeOC<sub>6</sub>H<sub>4</sub>CH<sub>2</sub>N(O):NPh (IV), slightly yellowish solid, decomposed at 176-7° [ $\lambda_{\text{maximum}}$  250 m $\mu$  (log  $\epsilon$  4.104) in Et<sub>2</sub>O, infrared absorption bands at 6.60, 6.76, and 7.84 in Nujol compensated, 6.61, 6.77, 6.72, 7.68, 7.85, and 7.95 in Nujol uncompensated, 7.69 in CHCl<sub>3</sub>.]. IV refluxed 2 hrs. with PhNCO in C<sub>6</sub>H<sub>6</sub> or with EtI in C<sub>6</sub>H<sub>6</sub> was recovered unchanged. IV (1.2 g.) refluxed 0.5 hr. with 0.38 g. powdered LiAlH<sub>4</sub> in 30 cc. tetrahydrofuran (V), the mixture decomposed with ice, the inorg. precipitate filtered off, washed with Et<sub>2</sub>O, the combined Et<sub>2</sub>O solution

dried

with MgSO<sub>4</sub>, evaporated in vacuo, the residue treated with hexane, and the resulting yellowish solid (0.62 g.) extracted with MeOH gave from the solution II, m. 119-20°; the MeOH-insol. material, recrystd. several times from EtOAc-EtOH, gave 0.051 g. p-MeOC<sub>6</sub>H<sub>4</sub>CH:NNPhC(:NNHPh)C<sub>6</sub>H<sub>4</sub>OMe-p (VI),

colorless crystals, m. 195-7°. II (1.13 g.) refluxed 2 hrs. with 0.38 LiAlH<sub>4</sub> in 50 cc. V, the mixture worked up in the usual way, and the yellow, gummy residue treated with MeOH left 0.08 g. VI, m. 197-8° (from EtOAc-EtOH); evaporation of the MeOH solution gave 0.73 g. II. III (1.21 g.) refluxed 2 hrs. with 1.8 g. LiAlH<sub>4</sub> in 75 cc. dry Et<sub>2</sub>O, the mixture decomposed with wet Et<sub>2</sub>O, the precipitated inorg. salts extracted continuously 24 hrs. with 100 cc. Et<sub>2</sub>O, and the combined Et<sub>2</sub>O solution dried with MgSO<sub>4</sub> and evaporated in vacuo gave 0.95 g. brown residue which, extracted with MeOH, yielded 0.03 g. III; from the MeOH solution was isolated I, m. 155-7° (from hexane), infrared absorption bands at 3.02, 6.24, 6.68, 6.93, 7.395, 7.79, and 6.355  $\mu$ . I (1.96 g.) and 0.4 g. LiAlH<sub>4</sub> in 50 cc. V refluxed 3 hrs., the mixture worked up in the usual way, and the resulting oily residue (2 g.) treated with small portions of pentane gave 1.74 g. I, m. 155-6° (from hexane); the pentane washing extracted with aqueous NaHSO<sub>3</sub> saturated with solid KOH, the alkaline extract extracted with 100 cc. Et<sub>2</sub>O, and the Et<sub>2</sub>O extracted dried and evaporated in vacuo gave 0.08 g. oily residue smelling strongly of BzH. Al strips (2 g.) amalgamated with 5% HgCl<sub>2</sub>, washed with MeOH and Et<sub>2</sub>O, refluxed 0.5 min. with 200 mg. III in 40 cc. Et<sub>2</sub>O, then 5 hrs. with stirring after addition of 1.5 cc. H<sub>2</sub>O, let stand overnight, the precipitated Al salts filtered off, washed with 90 cc. Et<sub>2</sub>O, the combined Et<sub>2</sub>O solution dried, evaporated in vacuo, and the residual white solid (89 mg.), m. 150-2°, recrystd. from cyclohexane-MeOAc gave a compound C<sub>14</sub>H<sub>14</sub>N<sub>2</sub>, colorless prisms, m. 154.5-5.5°, infrared absorption bands at 3.04, 6.25, 6.72, 6.91, 7.43, 7.72; from the precipitated Al salts with 80 cc. boiling CHCl<sub>3</sub> 52 mg. III was extracted. To EtMgI from 3.12 g. EtI and 0.5 g. Mg in 50 cc. Et<sub>2</sub>O was added 1.86 g. III suspended in 70 cc. hot C<sub>6</sub>H<sub>6</sub>, the mixture refluxed 0.5 hr. with stirring, the Et<sub>2</sub>O distilled off, the residue refluxed 1 hr., the solution hydrolyzed with ice and NH<sub>4</sub>Cl, extracted with 150 cc. Et<sub>2</sub>O, the extract dried, evaporated in vacuo, and the semisolid residue (1.32 g.) dissolved in 60 cc. C<sub>6</sub>H<sub>6</sub>; 5 cc. solution chromatographed on Al<sub>2</sub>O<sub>3</sub> showed the presence of at least 5 colored substances; 5 cc. solution treated with 2,4-(O<sub>2</sub>N)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>NHNH<sub>2</sub> in MeOH and H<sub>2</sub>SO<sub>4</sub> gave 2,4-(O<sub>2</sub>N)<sub>2</sub>C<sub>6</sub>H<sub>3</sub>NHN:CEtPh, m. 187-90° with sintering at 168° (from cyclohexane-C<sub>6</sub>H<sub>6</sub>); 20 cc. solution extracted with 35 cc. N HCl, the extract treated with Darco, cooled in ice, **neutralized** with 20% aqueous NaOH, extracted with Et<sub>2</sub>O, and the Et<sub>2</sub>O extract dried with K<sub>2</sub>CO<sub>3</sub> and evaporated in vacuo gave 240 mg. (169 mg. after several crystns. from cyclohexane, corresponding to a min. of 29%) IIIA, white crystals, m. 113-15°, infrared absorption bands at 2.81, 2.95, 6.08, 6.30, 6.33, 6.74, 6.91, 7.34, 8.14, 9.75, 12.02  $\mu$ , HCl salt, m. 214-18° (from EtOH-C<sub>6</sub>H<sub>6</sub>). I (200 mg.) in 10 cc. dry C<sub>6</sub>H<sub>6</sub> agitated under O until 23 cc. had been taken up, the solution hydrogenated under atmospheric pressure over 50 mg. Pt catalyst (prereduced in 7 cc. EtOAc), the mixture filtered, the filtrate evaporated, and the residue dissolved in pentane containing the min. amount of EtOAc to effect solution and cooled gave 0.123 g. BzNHNHPh, m. 170-1° (from cyclohexane-CHCl<sub>3</sub>). PhCH:NNMePh (1.24 g.) and 0.3 g. LiAlH<sub>4</sub> refluxed 3 hrs. in 40 cc. V gave 0.83 g. yellow oil; a portion treated with HCl in Et<sub>2</sub>O gave a HCl salt which was obtained by very slow crystallization from cyclohexane-C<sub>6</sub>H<sub>6</sub> in 2 forms of crystals, chunky plates and long silky needles, both m. 107-14°; the plates seemed to change to needles before melting, and also in contact with the needles in a solvent. The free **base** showed infrared absorption bands at 6.25, 6.39, 6.68, 6.88, 7.25, 7.57, 7.66, 8.43, 8.99

μ. III (1.021 g.) in 100 cc. glacial AcOH hydrogenated 24 hrs. over 0.301 g. 10% Pd-C, the mixture filtered, the filtrate diluted with an equal volume of H<sub>2</sub>O, carefully saturated with Na<sub>2</sub>CO<sub>3</sub> while being cooled in ice-salt, extracted with Et<sub>2</sub>O, the extract dried, evaporated in vacuo, and the residue dissolved in 30 cc. 80% hexane-C<sub>6</sub>H<sub>6</sub>, chromatographed on Al<sub>2</sub>O<sub>3</sub>, and eluted with 100 cc. 80% C<sub>6</sub>H<sub>6</sub>-hexane gave 0.0724 g. unidentified **base**, colorless oil with a strong lemonlike odor which yielded a HCl salt, m. 213-15°. The **base** gave with PhNCO a phenylthiourea derivative, m. 144-5° (sublimed). The **base** showed infrared absorption bands at 2.95, 3.41, 3.51, 6.23, 6.65, 6.83, 6.98, 7.57, 7.95, 8.50, and 14.40 μ in CHCl<sub>3</sub>. The HCl salt showed infrared bands at 2.92, 3.71, 3.98, 6.21, 6.32, 6.67, 6.75, 6.87, 7.31 μ in CHCl<sub>3</sub>. The column washed with C<sub>6</sub>H<sub>6</sub> and CHCl<sub>3</sub> and eluted with 200 cc. 95% EtOH gave 0.1973 g. of a basic compound C<sub>26</sub>H<sub>24</sub>N<sub>4</sub>O<sub>3</sub>, colorless crystals, m. 119-20° with sintering at 117° (sublimed in vacuo and recrystd. from Et<sub>2</sub>O), depressing the m.p. of IIIA to 87-110°; infrared absorption bands at 2.98, 3.05, 5.92, 6.23, 6.36, 6.70, 6.88, 7.67, 14.25 μ in CHCl<sub>3</sub>; HCl salt, m. 220-2°; picrate, bright yellow crystalline powder, m. 239-41° (from Me<sub>2</sub>CO); the parent **base** of the picrate corresponds to "C<sub>11</sub>H<sub>13</sub>N<sub>2</sub>O<sub>2</sub> or C<sub>22</sub>H<sub>26</sub>N<sub>4</sub>O<sub>4</sub>" (the nature of this discrepancy has not yet been investigated). To 0.222 g. PtO<sub>2</sub> in 20 cc. glacial AcOH prereduced with 271 cc. H was added 1.204 g. IV, the mixture stirred at room temperature and atmospheric pressure 20 hrs. under H, filtered, diluted with 10 cc. 6N HCl and 30 cc. H<sub>2</sub>O, washed with Et<sub>2</sub>O, the acidic solution cooled in ice, saturated with solid KOH, extracted with three 50-cc. portions of Et<sub>2</sub>O, the Et<sub>2</sub>O extract washed with 5 cc. H<sub>2</sub>O, concentrated to 40 cc., extracted with 12 consecutive 2-cc. portions of 0.1N HCl and one 2-cc. portion of H<sub>2</sub>O, and each fraction let dry in a vacuum desiccator over KOH to give 30-40 mg. HCl salts with m.ps. varying from 165-80° to 178-97°; the remaining Et<sub>2</sub>O solution dried and evaporated in vacuo gave 0.4 g. basic residue yielding a HCl salt, m. 210-18° (decomposition).

CC 10 (Organic Chemistry)

IT **Oxidation**  
(mechanism of)

IT Spectra  
(of hydrazone **oxidation** products)

IT Hydrazones  
(**oxidation** and autoxidation of)

IT 305-30-6, Benamidine, N-phenyl-, hydrochloride 532-96-7, Benzoic acid, 2-phenylhydrazide 618-40-6, Hydrazine, 1-methyl-1-phenyl- 637-03-6, Benzene, arsenoso- 1527-91-9, Benamidine, N-phenyl- 1833-18-7, Hydrazine, 1-p-anisoyl-2-p-methoxybenzylidene-1-phenyl-, phenylhydrazone 2213-43-6, Piperidine, 1-amino- 2215-16-9, Arsenic, bis(diphenyl-), oxide 3375-37-9, Propiophenone, 2,4-dinitrophenylhydrazone 4095-46-9, Benzenearsonous acid, p-nitro- 4406-71-7, Benzeneazoxymethane, 1'-phenyl- 95982-57-3, Anisole, p-(phenylazoxymethyl)- 102395-95-9, Toluene, p-arsenoso- 688064-37-1, Benzene, 1-arsenoso-4-bromo- 778604-16-3, Benzene, 2-arsenoso-1,3,5-tribromo-  
(preparation of)

L10 ANSWER 8 OF 16 WPIX COPYRIGHT 2005 THE THOMSON CORP on STN

ACCESSION NUMBER: 2005-029399 [03] WPIX

CROSS REFERENCE: 2004-624825 [60]

DOC. NO. CPI: C2005-009296

TITLE: Production of controllable yields of combination of

products from phenol, methyl ethyl ketone, or acetone, comprises feeding **oxidation** feed having **alkylbenzene(s)** to **oxidation** reactor to produce **oxidation** mixture.

DERWENT CLASS: A41 E14 E17

INVENTOR(S): BLACK, J R; BUECHELE, J L; YANG, J

PATENT ASSIGNEE(S): (BLAC-I) BLACK J R; (BUEC-I) BUECHELE J L; (YANG-I) YANG J

COUNTRY COUNT: 1

PATENT INFORMATION:

PATENT NO	KIND	DATE	WEEK	LA	PG
US 2004236152	A1	20041125	(200503)*		28

APPLICATION DETAILS:

PATENT NO	KIND	APPLICATION	DATE
US 2004236152	A1 Provisional	US 2003-447526P	20030214
		US 2004-761676	20040121

PRIORITY APPLN. INFO: US 2003-447526P 20030214; US  
2004-761676 20040121

AN 2005-029399 [03] WPIX

CR 2004-624825 [60]

AB US2004236152 A UPAB: 20050112

NOVELTY - Controllable yields of a combination of products from phenol, methyl ethyl ketone (MEK), or acetone is produced by feeding an **oxidation** feed having **alkylbenzene(s)** to an **oxidation** reactor to produce an **oxidation** mixture. The **alkylbenzene(s)** is s-butylbenzene, or combination of s-butylbenzene and **cumene** at a weight ratio of **cumene** to s-butylbenzene.

DETAILED DESCRIPTION - Production of controllable yields of a combination of products from phenol, MEK, or acetone, comprises feeding an **oxidation** feed having **alkylbenzene(s)** from s-butylbenzene or combination of s-butylbenzene and **cumene** at a weight ratio of **cumene** to s-butylbenzene to an **oxidation** reactor to produce an **oxidation** mixture; exposing the **oxidation** mixture to **oxidation** conditions to produce an **oxidation** product stream having product **hydroperoxides** from s-butylbenzene **hydroperoxide** or combination of s-butylbenzene **hydroperoxide** and **cumene hydroperoxide**; cleaving the product **hydroperoxides** under cleavage conditions to produce a cleavage product having combination from phenol and MEK, or phenol, acetone, and MEK; separating the cleavage product under separation conditions to separate a crude phenol fraction having phenol and a crude ketone stream from crude MEK stream or crude acetone/MEK stream comprising MEK and acetone; and recovering product(s) from MEK product or combination comprising an MEK product and an acetone product.

INDEPENDENT CLAIMS are also included for:

(1) processes for producing controllable yields of a combination of products selected from:

- (a) phenol and methyl ethyl ketone (MEK); and
- (b) phenol and MEK and acetone; and

(2) a process for producing phenol, methyl ethyl ketone and acetone.  
USE - For producing controllable yields of a combination of products from phenol, MEK, or acetone.

ADVANTAGE - The invention produces controllable yields of phenol, MEK, and acetone during the manufacture of phenol depending on the market demand.

DESCRIPTION OF DRAWING(S) - The figure is a block diagram of the production of controllable yields of a combination of products.  
Dwg.1/5

L10 ANSWER 9 OF 16 WPIX COPYRIGHT 2005 THE THOMSON CORP on STN  
ACCESSION NUMBER: 2003-877181 [81] WPIX  
DOC. NO. CPI: C2003-247741  
TITLE: Preparation of substituted pyridinylmethyl-sulfinyl-benzimidazole, useful in treating ulcers, comprises **oxidation** of a pyridinylmethyl prochiral sulfide derivative of benzimidazole in the presence of a **base** and a catalyst.  
DERWENT CLASS: B02  
INVENTOR(S): CHHABADA, V C; PATEL, V M; REHANI, R B; SONI, R R; THENNATI, R  
PATENT ASSIGNEE(S): (SUNP-N) SUN PHARM IND LTD  
COUNTRY COUNT: 102  
PATENT INFORMATION:

PATENT NO	KIND	DATE	WEEK	LA	PG
WO 2003089408	A2	20031030	(200381)*	EN	31
RW: AT BE BG CH CY CZ DE DK EA EE ES FI FR GB GH GM GR HU IE IT KE LS LU MC MW MZ NL OA PT RO SD SE SI SK SL SZ TR TZ UG ZM ZW					
W: AE AG AL AM AT AU AZ BA BB BG BR BY BZ CA CH CN CO CR CU CZ DE DK DM DZ EC EE ES FI GB GD GE GH GM HR HU ID IL IN IS JP KE KG KP KR KZ LC LK LR LS LT LU LV MA MD MG MK MN MW MX MZ NO NZ OM PH PL PT RO RU SC SD SE SG SK SL TJ TM TN TR TT TZ UA UG US UZ VC VN YU ZA ZM ZW					
AU 2003262375	A1	20031103	(200438)		

## APPLICATION DETAILS:

PATENT NO	KIND	APPLICATION	DATE
WO 2003089408	A2	WO 2003-IN164	20030421
AU 2003262375	A1	AU 2003-262375	20030421

## FILING DETAILS:

PATENT NO	KIND	PATENT NO
AU 2003262375	A1 Based on	WO 2003089408

PRIORITY APPLN. INFO: IN 2002-MU365 20020422; IN  
2002-MU299 20020422

AN 2003-877181 [81] WPIX

AB WO2003089408 A UPAB: 20031216

NOVELTY - Preparation of substituted pyridinylmethyl-sulfinyl-benzimidazole (I) or their salts comprises enantioselective catalytic **oxidation** of a substituted pyridinylmethyl prochiral sulfide derivative of benzimidazole with an oxidizing agent in an organic solvent

in the presence of a **base** and a catalyst comprising titanium or vanadium complexed with a chiral monodentate ligand.

DETAILED DESCRIPTION - Preparation of an optically active enantiomer or enantiomerically enriched form of a substituted pyridinylmethylsulfinyl-benzimidazole compound of formula (I) comprises the enantioselective catalytic **oxidation** of a substituted pyridinylmethyl prochiral sulfide derivative of benzimidazole of formula (II) with an oxidizing agent in an organic solvent in the presence of a **base** and a catalyst that may be titanium or vanadium complexed with a chiral monodentate ligand.

R1-R4 = H, (1-4)C alkyl, (1-4)C alkoxy, aryl(oxy), halo or alkoxy substituted analogs.

An INDEPENDENT CLAIM is also included for a process for purification of alkali or alkaline earth metal salts of the S-enantiomer of 5-methoxy-2-((4-methoxy-3,5-dimethyl-2-pyridinylmethyl)sulfinyl)-1H-benzimidazole (IV), which comprises the treatment of an alkali or alkaline earth metal salt having a sulfone impurity with a solvent system containing an organic solvent (ketone or nitrile) and isolation of (IV) which is substantially free of sulfone impurity.

ACTIVITY - Antiulcer.

MECHANISM OF ACTION - Proton pump inhibitor.

USE - The enantioselective catalytic **oxidation** process provides a convenient method for the preparation of an optically active enantiomer or enantiomerically enriched form of (I), specifically sulfoxides of omeprazole, pantoprazole, rabeprazole and lansoprazole, which are proton pump inhibitors useful in the treatment of ulcers. The process also enables the preparation of optically active alkali or alkaline earth metal salts of (I) and the purification of alkali or alkaline earth metal salts of (IV).

ADVANTAGE - The enantioselective catalytic **oxidation** preparation of (I) is a facile, convenient and inexpensive process, utilizing a diverse pool of reagents to achieve optical purity. It provides enantiomeric excess greater than 98%. Preparation of alkali and/or alkaline earth metal salts of (IV) is also a simple and easy process without the need to separate the unwanted isomer and provides a product substantially free of impurity (less than 0.2%).

Dwg. 0/0

L10 ANSWER 10 OF 16 WPIX COPYRIGHT 2005 THE THOMSON CORP on STN

ACCESSION NUMBER: 2003-644960 [61] WPIX

CROSS REFERENCE: 2003-066387 [06]

DOC. NO. CPI: C2003-176300

TITLE: Preparation of **cumene hydroperoxide**, useful in acid-catalyzed cleavage to phenol and acetone, involves reaction of **cumene** and oxygen in the presence of an aqueous phase containing **ammonia** but no alkali(ne earth) metal.

DERWENT CLASS: E14

INVENTOR(S): FULMER, J W; KIGHT, W D; SCOTT, E E

PATENT ASSIGNEE(S): (GEEL-N) GEN ELECTRONIC CO; (GENE) GENERAL ELECTRIC CO

COUNTRY COUNT: 1

PATENT INFORMATION:

PATENT NO	KIND	DATE	WEEK	LA	PG
US 2003092943	A1	20030515	(200361)*		9
US 6620974	B2	20030916	(200362)		

## APPLICATION DETAILS:

PATENT NO	KIND	APPLICATION	DATE
US 2003092943	A1 Div ex	US 2001-916775	20010727
		US 2002-225095	20020821
US 6620974	B2 Div ex	US 2001-916775	20010727
		US 2002-225095	20020821

## FILING DETAILS:

PATENT NO	KIND	PATENT NO
US 2003092943	A1 Div ex	US 6465695
US 6620974	B2 Div ex	US 6465695

PRIORITY APPLN. INFO: US 2001-916775 20010727; US  
2002-225095 20020821

AN 2003-644960 [61] WPIX

CR 2003-066387 [06]

AB US2003092943 A UPAB: 20030928

NOVELTY - Preparation of **cumene hydroperoxide** involves reaction of **cumene** and oxygen in the presence of a water phase containing aqueous **ammonia**; and in the absence of an additive containing an alkali or alkaline earth metal.

DETAILED DESCRIPTION - INDEPENDENT CLAIMS are also included for the following:

(a) a system (S1) comprising a device for reacting **cumene** with oxygen in the presence of ammonium hydroxide, and in the absence of an additive containing an alkali(ne earth) metal;

(b) a system (S2) comprising a **cumene** feed in fluid communication with a reactor having a **cumene hydroperoxide** outlet; an oxygen feed in fluid communication with the reactor; and an ammonium hydroxide feed in fluid communication with the **cumene** feed and/or the reactor. The **cumene** feed, the oxygen feed, the ammonium hydroxide feed and the reactor are free of an additive containing alkali(ne earth) metal.

USE - **Cumene hydroperoxide** is useful in a number of application e.g. acid-catalyzed cleavage to phenol and acetone.

ADVANTAGE - Using free **ammonia** as the **neutralizing** agent eliminates the need for alkali(ne earth) salt or **base** additives (e.g. Group IA or IIA metal carbonates, phosphates, hydroxides or hydrates). The method improves pH control, reduces phenol inhibitor formation, lowers initial plant investment, gives high plant on-stream factor, increases production rate, and eliminates complex systems and expensive equipment.

Dwg. 0/2

L10 ANSWER 11 OF 16 WPIX COPYRIGHT 2005 THE THOMSON CORP on STN

ACCESSION NUMBER: 2002-733047 [79] WPIX

DOC. NO. CPI: C2002-207534

TITLE: Preparation of des-methyl cyproheptadine useful as an intermediate for e.g. anti-plasma medicine, anti-allergy medicine and antihistamine medicine.

DERWENT CLASS: B03

INVENTOR(S): TATARA, A; YATAGAI, M

PATENT ASSIGNEE(S): (AJIN) AJINOMOTO CO INC

COUNTRY COUNT: 100



## PATENT INFORMATION:

PATENT NO	KIND	DATE	WEEK	LA	PG
WO 2002081450	A1	20021017	(200279)*	JA	21
RW: AT BE CH CY DE DK EA ES FI FR GB GH GM GR IE IT KE LS LU MC MW MZ NL OA PT SD SE SL SZ TR TZ UG ZM ZW					
W: AE AG AL AM AT AU AZ BA BB BG BR BY BZ CA CH CN CO CR CU CZ DE DK DM DZ EC EE ES FI GB GD GE GH GM HR HU ID IL IN IS JP KE KG KP KR KZ LC LK LR LS LT LU LV MA MD MG MK MN MW MX MZ NO NZ OM PH PL PT RO RU SD SE SG SI SK SL TJ TM TN TR TT TZ UA UG US UZ VN YU ZA ZM ZW					
AU 2002243024	A1	20021021	(200433)		

## APPLICATION DETAILS:

PATENT NO	KIND	APPLICATION	DATE
WO 2002081450	A1	WO 2002-JP3294	20020402
AU 2002243024	A1	AU 2002-243024	20020402

## FILING DETAILS:

PATENT NO	KIND	PATENT NO
AU 2002243024	A1 Based on	WO 2002081450

PRIORITY APPLN. INFO: JP 2001-103266 20010402

AN 2002-733047 [79] WPIX

AB WO 2002081450 A-UPAB: 20021209

NOVELTY - Preparation of des-methyl cyproheptadine (3) comprises reacting cyproheptadine (I) with an **oxidation** agent to obtain the N-oxide (2) and then reacting with divalent Fe salt.

DETAILED DESCRIPTION - Preparation of des-methyl cyproheptadine of formula (3) comprises reacting cyproheptadine of formula (I) with an **oxidation** agent to obtain the N-oxide of formula (2) and then reacting with divalent Fe salt.

An INDEPENDENT CLAIM is also included for a method of refining des-methyl cyproheptadine by adding acid in the reaction solution containing (2) and divalent Fe salt, condensing or cooling solvent to precipitate crystalline (3) under acidic conditions, and then further **neutralizing** the **base** to precipitate crystalline (3).

USE - Used as an intermediate medicine for cellotonin (sic) antagonistic, anti-plasma medicine, anti-allergy medicine and antihistamine medicine.

ADVANTAGE - The preparation does not generate alkyl halide which is difficult to decompose. The yield is high.  
Dwg.0/0

L10 ANSWER 12 OF 16 WPIX COPYRIGHT 2005 THE THOMSON CORP on STN

ACCESSION NUMBER: 2002-454310 [48] WPIX

DOC. NO. CPI: C2002-129067

TITLE: Preparation of oligomeric compounds used in antiviral therapy by coupling deprotected 5'-O-protected compound with activated phosphorous to form extended compound, followed by treatment with oxidizing and capping agent.

DERWENT CLASS: A96 B04 D16

INVENTOR(S): SANGHVI, Y S; SONG, Q

PATENT ASSIGNEE(S): ~~ISIS-N~~ ISIS PHARM INC  
 COUNTRY COUNT: 97  
 PATENT INFORMATION:

PATENT NO	KIND	DATE	WEEK	LA	PG
WO 2002014340	A1	20020221	(200248)*	EN	95
RW: AT BE CH CY DE DK EA ES FI FR GB GH GM GR IE IT KE LS LU MC MW MZ NL OA PT SD SE SL SZ TR TZ UG ZW					
W: AE AG AL AM AT AU AZ BA BB BG BR BY BZ CA CH CN CO CR CU CZ DE DK DM DZ EC EE ES FI GB GD GE GH GM HR HU ID IL IN IS JP KE KG KP KR KZ LC LK LR LS LT LU LV MA MD MG MK MN MW MX MZ NO NZ PL PT RO RU SD SE SG SI SK SL TJ TM TR TT TZ UA UG US UZ VN YU ZA ZW					
AU 2001081275	A	20020225	(200248)		
EP 1311526	A1	20030521	(200334)	EN	
R: AL AT BE CH CY DE DK ES FI FR GB GR IE IT LI LT LU LV MC MK NL PT RO SE SI TR					
US 2004198972	A1	20041007	(200466)		
US 6809195	B1	20041026	(200470)		

## APPLICATION DETAILS:

PATENT NO	KIND	APPLICATION	DATE
WO 2002014340	A1	WO 2001-US25623	20010816
AU 2001081275	A	AU 2001-81275	20010816
EP 1311526	A1	EP 2001-959753	20010816
		WO 2001-US25623	20010816
US 2004198972	A1 Cont of	US 2000-640279	20000816
		US 2004-828659	20040421
US 6809195	B1	US 2000-640279	20000816

## FILING DETAILS:

PATENT NO	KIND	PATENT NO
AU 2001081275	A Based on	WO 2002014340
EP 1311526	A1 Based on	WO 2002014340

PRIORITY APPLN. INFO: US 2000-640279 20000816; US  
 2004-828659 20040421

AN 2002-454310 [48] WPIX

AB WO 200214340 A UPAB: 20020730

NOVELTY - Preparation of oligomeric compounds (I) comprises coupling a deprotected 5'-O-protected compound with an activated phosphorous composition (III) to form an extended compound (IV), followed by treatment with an oxidizing and capping agent.

DETAILED DESCRIPTION - Preparation of oligomeric compounds (I) having at least one group of formula (i) or (ii) comprises deprotecting a 5'-O-protected compound of formula (II) or (III) with a deprotecting agent, coupling the deprotected (II) or (III) with an activated phosphorous composition of formula (IV) to form an extended compound of formula (V) or (VI) and treating (V) or (VI) with a mixture of an oxidizing and capping agent to form the oligomeric compound.

X2 = O or S;

X1 = Pg-O, Pg-S-, 1-10C alkyl, CH<sub>3</sub>(CH<sub>2</sub>)<sub>nn</sub>-O-, R<sub>2</sub>R<sub>3</sub>N- or a group remaining from coupling a chiral auxiliary;  
 nn = 0-10;

Pg = CH<sub>3</sub>, -CH<sub>2</sub>CH<sub>2</sub>CN, -C(CH<sub>3</sub>)(CH<sub>3</sub>)-CCl<sub>3</sub>, -CH<sub>2</sub>-CCl<sub>3</sub>, -CH<sub>2</sub>CH=CH<sub>2</sub>, CH<sub>2</sub>CH<sub>2</sub>SiCH<sub>3</sub>, 2-yl-ethyl phenylsulfonate, delta -cyanobutenyl, cyano para-xylyl, diphenylsilylethyl, 4-nitro-2-yl-ethylbenzene, 2-yl-ethyl-methyl sulfonate, methyl-N-trifluoroacetyl ethyl, acetoxy phenoxy ethyl or a blocking group (preferably acid stable and base labile blocking group);

R<sub>2</sub>, R<sub>3</sub> = H, 1-10C alkyl, cycloalkyl or aryl, or

NR<sub>2</sub>R<sub>3</sub> = cyclic group;

Bx = heterocyclic base group;

R<sub>1</sub> = H, blocked hydroxyl group or a sugar substituent group;

T<sub>1</sub> = hydroxy protecting group;

~~T<sub>2</sub> = a covalent bond to a support media, a nucleoside bound to a support media, a nucleotide, oligonucleoside or oligonucleotide;~~

T<sub>3</sub> = OH protecting group, nucleoside, nucleotide, oligonucleoside or oligonucleotide;

R<sub>4</sub> = N(L<sub>1</sub>)L<sub>2</sub>;

L<sub>1</sub>, L<sub>2</sub> = 1-6C alkyl or 5-7C cyclic aliphatic ring, or

L<sub>1</sub> + L<sub>2</sub> = 4-13 membered heterocyclyl including the N atom to which L<sub>1</sub> and L<sub>2</sub> are attached, and

R<sub>5</sub> = X<sub>1</sub> or

PR<sub>4</sub>R<sub>5</sub> = a chiral auxiliary.

An INDEPENDENT CLAIM is included for a synthetic process (S<sub>1</sub>) which comprises adding methylamine, carbon disulfide and an organic solvent to a basic aqueous solution, to form a mixture, adding ice and acid (preferably glacial acetic acid) to the mixture, to form an acidified mixture, adding an oxidizing agent to the acidified mixture, to form an oxidized mixture, adding a non-polar solvent to the oxidized mixture to form a precipitate, and isolating and washing the precipitate with aqueous acid and a non-polar organic solvent.

USE - (I) Are useful in molecular biological research and antiviral therapy and as antisense agents.

~~ADVANTAGE~~ The oxidation and capping steps are combined into a single step, which improves the efficiency of synthesis. The overall synthesis is therefore completed in less time with a reduction in bulk reagents required. Thus the methods result in increased efficiency and are especially amenable to the large-scale synthesis of oligomeric compounds.

Dwg.0/0

L10 ANSWER 13 OF 16 WPIX COPYRIGHT 2005 THE THOMSON CORP on STN  
ACCESSION NUMBER: 2003-370765 [35] WPIX  
DOC. NO. CPI: C2003-098182  
TITLE: Preparation of (2,8-bis(trifluoromethyl)-4-quinoliny)-2-pyridinylmethanone used as mefloquine intermediate by condensing haloquinoline with alpha-picolyl derivative in presence of organic solvent, base and phase transfer catalyst.  
DERWENT CLASS: B02  
INVENTOR(S): CHAWLA, H P S; JOHAR, P S; MEENA, R A; MITTAL, A; NEGI, V S  
PATENT ASSIGNEE(S): (NAPH-N) NAT PHARM EDUCATION & RES SOC; (NAPH-N) NAT INST PHARM EDUCATION & RES  
COUNTRY COUNT: 2  
PATENT INFORMATION:

PATENT NO	KIND DATE	WEEK	LA	PG
US 2002188129	A1 20021212 (200335)*			10

CN 1370777 A 20020925 (200335)  
US 6500955 B1 20021231 (200335)

## APPLICATION DETAILS:

PATENT NO	KIND	APPLICATION	DATE
US 2002188129	A1	US 2002-58304	20020130
CN 1370777	A	CN 2002-103180	20020204
US 6500955	B1	US 2002-58304	20020130

PRIORITY APPLN. INFO: IN 2001-DE129 20010202

AN 2003-370765 [35] WPIX

AB US2002188129 A UPAB: 20031030

NOVELTY - Preparation of (2,8-bis(trifluoromethyl)-4-quinolinyl)-2-pyridinylmethanone (I) comprises condensing a halo-quinoline with an alpha-picolyl derivative in the presence of an organic solvent, a **base** and a phase transfer catalyst at -10 - 90 deg. C, adding oxidizing agent, cooling and removing solvent.

DETAILED DESCRIPTION - Preparation of (2,8-bis(trifluoromethyl)-4-quinolinyl)-2-pyridinylmethanone (I) comprises:

(1) condensing a halo-quinoline (a) with an alpha-picolyl derivative (b) in the presence of an organic solvent (s1), a **base** and a phase transfer catalyst (c) at -10 - 90 deg. C;

(2) adding an oxidizing agent (d) to the reaction mixture containing (2,8-bis-trifluoromethyl-quinolin-4-yl)-pyridin-2-yl-acetonitrile (II) at -10 - 90 deg. C;

(3) cooling the mixture and **neutralizing** with acid (e) followed by extraction with an organic solvent (s2); and

(4) removing the organic solvent and crystallizing (I).

USE - Used as a drug intermediate for preparing antimalarial drug mefloquine.

ADVANTAGE - (c) Can transfer the carbon ion generated from pyridylacetonitrile or its analogues. (d) Acts as a nucleophile agent. A one pot, single step, simple and economical process is used for the preparation of (II) without the use of hazardous chemicals. The method eliminates the use of expensive anhydrous solvents or hazardous reagents. The process does not isolate (II) before **oxidation**.  
Dwg.0/4

L10...ANSWER 14 OF 16...WPIX...COPYRIGHT 2005 THE THOMSON CORP on STN..

ACCESSION NUMBER: 2003-352106 [33] WPIX

DOC. NO. CPI: C2003-092667

TITLE: Conversion of carbonyl-type impurities in phenol to high-boiling derivatives, involves contacting the phenol with a catalyst of layered double hydroxide composition containing divalent and trivalent metals.

DERWENT CLASS: A41 E14

INVENTOR(S): FULMER, J W; HASYAGAR, U; KUMBHAR, P; SINGH, B; TATAKE, P  
A

PATENT ASSIGNEE(S): (GENE) GENERAL ELECTRIC CO

COUNTRY COUNT: 101

PATENT INFORMATION:

PATENT NO	KIND	DATE	WEEK	LA	PG
US 6486365	B1	20021126 (200333)*			8

WO 2003084910 A1 20031016 (200378) EN  
 RW: AT BE BG CH CY CZ DE DK EA EE ES FI FR GB GH GM GR HU IE IT KE LS  
 LU MC MW MZ NL OA PT RO SD SE SI SK SL SZ TR TZ UG ZM ZW  
 W: AE AG AL AM AT AU AZ BA BB BG BR BY BZ CA CH CN CO CR CU CZ DE DK  
 DM DZ EC EE ES FI GB GD GE GH GM HR HU ID IL IN IS JP KE KG KP KR  
 KZ LC LK LR LS LT LU LV MA MD MG MK MN MW MX MZ NO NZ OM PH PL PT  
 RO RU SD SE SG SK SL TJ TM TN TR TT TZ UA UG UZ VN YU ZA ZM ZW  
 AU 2003218136 A1 20031020 (200436)  
 EP 1494985 A1 20050112 (200504) EN  
 R: AL AT BE BG CH CY CZ DE DK EE ES FI FR GB GR HU IE IT LI LT LU LV  
 MC MK NL PT RO SE SI SK TR  
 KR 2004106318 A 20041217 (200525)

## APPLICATION DETAILS:

PATENT NO	KIND	APPLICATION	DATE
US 6486365	B1	US 2002-63258	20020404
WO 2003084910	A1	WO 2003-US7695	20030313
AU 2003218136	A1	AU 2003-218136	20030313
EP 1494985	A1	EP 2003-714126	20030313
		WO 2003-US7695	20030313
KR 2004106318	A	KR 2004-715821	20041004

## FILING DETAILS:

PATENT NO	KIND	PATENT NO
AU 2003218136	A1 Based on	WO 2003084910
EP 1494985	A1 Based on	WO 2003084910

PRIORITY APPLN. INFO: US 2002-63258 20020404

AN 2003-352106 [33] WPIX

AB US 6486365 B UPAB: 20030526

NOVELTY - A process for converting carbonyl-type impurities present in a phenolic solvent to high-boiling derivatives involves contacting phenolic solvent with a catalyst, at a catalytically-effective temperature, to produce a phenol-containing stream with high boiling derivatives and a reduced amount of carbonyl-type impurities. The catalyst comprises a layered double hydroxide composition or its hydrate.

DETAILED DESCRIPTION - A process for converting carbonyl-type impurities present in a phenolic solvent to high-boiling derivatives involves contacting the phenolic solvent with a catalyst, at a catalytically-effective temperature, to produce a phenol-containing stream with high boiling derivatives and a reduced amount of carbonyl-type impurities. The catalyst comprises a layered double hydroxide composition of the formula (I), or its hydrate.

$(MII1-xMIIIX(OH)_2)(An^-)_{x/n}$  (I)

MII = divalent metal cation;

MIIIX = trivalent metal cation;

A = interlayer anion of charge n-; and

x = 0.12-0.8.

INDEPENDENT CLAIMS are also included for:

- (1) a process for separating carbonyl-type impurities from a phenolic solvent, by converting the impurities as above and separating the high-boiling derivatives of the carbonyl-type impurities from the phenol-containing stream using conventional separation techniques;
- (2) conversion of **cumene** to phenol which involves producing

a crude phenol stream (CPS) (11) containing carbonyl-type impurities, and contacting CPS with the catalyst to produce a phenol product containing less carbonyl-type impurities; and

(3) facility for converting **cumene** to phenol, comprising:

(a) a vessel containing **cumene**;

(b) a reaction vessel (I) connected to this vessel, where the **cumene** is oxidized (2) to form a **cumene hydroperoxide** (CHP) mixture;

(c) a reaction vessel (II) connected to the reaction vessel (I), where the CHP mixture is cleaved (3) to form a crude cleavage mass mixture;

(d) a reaction vessel (III) connected to the reaction vessel (II), where a **base** is added to the crude cleavage mass mixture for **neutralization** (4);

(e) a separation section connected to receive the **neutralized** crude cleavage mass mixture, where the mixture is separated into streams, one of which is a CPS stream comprising phenol containing carbonyl-type impurities;

(f) a temperature control mechanism connected to receive the CPS; and

(g) a catalyst bed comprising the layered double hydroxide catalyst, to produce a purified phenol-containing product (17).

USE - Removing carbonyl-type impurities during production of phenol (claimed).

**ADVANTAGE** - The process can be applied in conventional industrial process for converting **cumene** to phenol without introducing significant amounts of additional contaminants such as 2MBF that can foul the machinery or increase the pollution levels. The catalyst can be regenerated.

**DESCRIPTION OF DRAWING(S)** - The figure shows a process of converting **cumene** into phenol.

**Cumene** 1

**Oxidation** 2

**Cleavage** 3

**Neutralization** 4

Distillation columns 6,8,10,15

Crude phenol stream 11

Hydrotalcite type material 12

Temperature control mechanism 13

Product phenol stream 17

Dwg.1/1

L10 ANSWER 15 OF 16 WPIX COPYRIGHT 2005 THE THOMSON CORP on STN  
ACCESSION NUMBER: 1983-42118K [18] WPIX  
DOC. NO. CPI: C1983-041037  
TITLE: Methyl phenol production from **alkylbenzene** tert.  
**hydroperoxide** - with catalytic hydrogenation of  
prim. **hydroperoxide** by-product.  
DERWENT CLASS: E14  
INVENTOR(S): COLVIN, H A  
PATENT ASSIGNEE(S): (GOOD) GOODYEAR TIRE & RUBBER CO  
COUNTRY COUNT: 7  
PATENT INFORMATION:

PATENT NO	KIND	DATE	WEEK	LA	PG
EP 77749	A	19830427	(198318)*	EN	17
R: DE FR IT					
JP 58079941	A	19830513	(198325)		

BR 8205957 A 19830913 (198343)  
 US 4431849 A 19840214 (198409)  
 CA 1188325 A 19850604 (198527)  
 EP 77749 B 19851218 (198551) EN  
 R: DE FR IT  
 DE 3268054 G 19860130 (198606)  
 JP 01049248 B 19891024 (198946)

## APPLICATION DETAILS:

PATENT NO	KIND	APPLICATION	DATE
JP 58079941	A	JP 1982-184406	19821020
US 4431849	A	US 1981-313517	19811021

PRIORITY APPLN. INFO: US 1981-313517 19811021

AN 1983-42118K [18] WPIX

AB EP 77749 A UPAB: 19930925

Production of a methylphenol (A) from the tert. **hydroperoxide** (B) present in an **oxidn.** mixture derived from **alkylbenzene** (I) (R is 3-4C sec. alkyl and n is 1-3) comprises hydrogenating the **oxidn.** mixture in inorganic acid medium over a Pd, Pt, Ni, Cr, Cu, Ru and Rh catalyst.

Pref. (B) is first rearranged in presence of acid catalyst, then the mixture hydrogenated (to reduce the amount of prim. **hydroperoxide** (c)), and **neutralised** with **base**. Hydrogenation is pref. at 0-200 (25-45) deg.C and 0-552 kPa for 0.2-10 hr.

(I) is pref. oxidised with air or oxygen, usually at 90-115 deg.C, then (A) rearranged conventionally. After hydrogenation the mixture is pref. **neutralised** with **ammonia** gas, then (A) and (I) recovered by distillation

The method is specified for production of p-cresol from p-cymene. It requires only low hydrogenation temperature; converts (c) back to reusable hydrocarbon starting material (avoiding a potentially explosive decomposition step) and gives a high-purity prod. No inorganic waste prods. are formed.

ABEQ EP 77749 B UPAB: 19930925

Prod. of a methylphenol (A) from the tert. **hydroperoxide** (B) present in an **oxidn.** mixt. derived from **alkylbenzene**

(I) (R is 3-4C sec. alkyl and n is 1-3) comprises hydrogenating the **oxidn.** mixt. in inorganic acid medium over a Pd, Pt, Ni, Cr, Cu, Ru and Rh catalyst.

Pref. (B) is first rearranged in presence of acid catalyst, then the mixt. hydrogenated (to reduce the amt. of prim. **hydroperoxide** (c)), and **neutralised** with **base**. Hydrogenation is pref. at 0-200 (25-45) deg.C and 0-552 kPa for 0.2-10 hr.

(I) is pref. oxidised with air or oxygen, usually at 90-115 deg.C, then (A) rearranged conventionally. After hydrogenation the mixt. is pref. **neutralised** with **ammonia** gas, then (A) and (I) recovered by distn.

The method is specified for prodn. of p-cresol from p-cymene. It requires only low hydrogenation temp.; converts (c) back to reusable hydrocarbon starting material (avoiding a potentially explosive decomposition step) and gives a high-purity prod. No inorganic waste prods. are formed.

ABEQ US 4431849 A UPAB: 19930925

Methyl phenol is prepd. from an **alkylbenzene** of formula (I), by

(a) contacting with O<sub>2</sub> to form an **oxidn.** prod. contg. tert. **hydroperoxide** and prim. **hydroperoxide**; (b) acid decomposing the **hydroperoxide** using a mineral acid catalyst; (c) hydrogenating at 0-200 deg. C and 0-552 kPa for 0.2-10 hrs. using Cr, Cu, Pd, Pt, Ni, Ru or Rh as a catalyst; (d) **neutralising** with NH<sub>3</sub>, NH<sub>4</sub>OH, alkali metal hydroxide or carbonate; and (e) recovering the ~~corresp. prod.~~ In the formula, R is a 3-4C sec. alkyl; and n is 1-3.

The process is esp. used to produce p-cresol from p-cymene, and the process is continuous or semi-continuous.

L10 ANSWER 16 OF 16 WPIX COPYRIGHT 2005 THE THOMSON CORP on STN  
 ACCESSION NUMBER: 1977-26998Y [15] WPIX  
 TITLE: Recovery of pure phenol in improved yield - from **cumene oxidn.** prod. minimising by prod. formation.  
 DERWENT CLASS: A41 E14  
 PATENT ASSIGNEE(S): (ALLC) ALLIED CHEM CORP  
 COUNTRY COUNT: 1  
 PATENT INFORMATION:

PATENT NO	KIND	DATE	WEEK	LA	PG
US 4016213	A	19770405	(197715)*		

PRIORITY APPLN. INFO: US 1971-139875 19710503

AN 1977-26998Y [15] WPIX

AB US 4016213 A UPAB: 19930901

An improvement is claimed in the process for obtaining phenol (I) from **cumene hydroperoxide** (II). (II) has been obtd. by liquid phase **oxidn.** of **cumene** (III) with molecular oxygen (IV), which involves (i) forming a reaction mixture by continuously feeding (III) **oxidn.** prod. containing at least 80 weight % (II) into a decomposer wherein incoming (II) is diluted by (II) decomposition prods. already there; (ii) maintaining reaction mixture at elevated temperature; (iii) introducing as decomposition catalyst (V) H<sub>2</sub>SO<sub>4</sub> or SO<sub>2</sub>; (iv) withdrawing reaction mixture from decomposer; (v) removing (V) from it; and (vi) fractionally distilling resultant prod. to separately recover acetone-, phenol and one or more by-product fractions.

The improvement comprises, in combination:- (a) conducting process in two decomposes linked serially, the first being equipped with an agitator, the second being tubular; (b) maintaining reaction mixture in decomposes at 75-95 degrees C; (c) feeding (V) to reaction mixture in first decomposer in amount = 0.002-0.02 weight % in (II)-feed; (d) conducting decomposition reaction at less than 0.3 weight % water on reaction mixture; (e) withdrawing reaction mixture from first decomposer at (II) concentration = 3-6 weight %, and from

second decomposer at (II) concentration = 3-6 weight %, and from second decomposer

at 0.3 weight % or less, on reaction mixture; and (f) adding a **base** (VI) to prod. withdrawn from second decomposer in excess of that to **neutralise** (V), and to adjust pH of prod. to from 6 to 8, wherein (VI) = alkali metal hydroxide or phenates, so that dehydration of dimethyl phenyl carbinol (VII) to alpha-methylstyrene (VIII) is substantially avoided during decomposition and distillation

(I) is produced from (III) in pure form in improved yield. Contamination of (I) with (VIII) is reduced, and formation of high b. pt.



by-prods. such as (VIII) dimer and cumyl phenol (IX) is reduced. In an example, crude **cumene hydroperoxide** was continuously decomposed by introduction into agitated, previously decomposed **cumene hydroperoxide** at 94 degrees C., with 19 minute hold-up time and 30 ppm H<sub>2</sub>SO<sub>4</sub>. 50% aqueous NaOH solution was added to the decomposition prod. mixture to pH=7, and fractionally distilled to separate acetone, phenol from the bottom.